



## ENCAPSULATED ORGANIC SOLUTION PARTICLES

### BACKGROUND OF THE INVENTION

Oils and other organic solutions of all viscosities are frequently the source of desirable active ingredients in the formulation of various compound products where the most utile or efficacious form is dry. Processes converting such viscous solutions into a dry condition including but not limited to; freeze-drying, spray-drying (either directly or after inclusion in an aqueous slurry), and dehydration, universally are destructive to the molecular structure of the desirable active ingredients including enzymes and fatty acid isomers all of which are heat sensitive.

Enzymes are protein or protein-based molecules that speed up chemical reactions in a living organism. An enzyme acts as catalyst for specific chemical reactions converting a specific set of reactants (called substrates) into specific products. Without enzymes, life as we know it, would not exist.

A fatty acid isomer (one of two or more molecules that are long chains of lipid-carboxylic acid found in fats and oils and in cell membranes as a component of phospholipids and glycolipids) is another class of desirable catalyst present in oils.

Spray drying is by definition the transformation of a feed from a liquid state (emulsion, dispersion, or paste) into a dried particulate form, achieved by spraying the liquid feed into a hot drying medium. It is a continuous or batch process consisting of several transformations including atomization, spray-air mixing, evaporation, and product separation.

Some such organic solutions, while having desirable qualities, have sensitivities to heat, light, and oxygen or, in the event of slurry-processing, reaction with other slurry components. The literature is replete with anecdotal product specific attempts to address unique conversions.

Each has limited scope and application. Further most methods have inherent or finite limitations as to the “load” or percent of active ingredient in the resulting dry compound.

For example, studies have linked long chain polyunsaturated fatty acids (PUFA) and especially omega-3 and omega-6 fatty acids that are contained in fish oil with multiple medicinal and nutritional functions. These include prevention of coronary heart disease, suppression of platelet-aggregation, decreasing the level of serum cholesterol, treatment of cerebral thrombosis myocardial infarction, as well as others. Thus, there is a desire on the part of the food industry to supplement foodstuffs with PUFA. This is normally done by incorporating an oil high in PUFA into the foodstuff. Fish oil is a main source for these oils, however, plant and microbial liquids are also sources of oils which are high in PUFA.

There are obstacles to the use of fish oils and to employing PUFA in foodstuffs. First, PUFAs are very sensitive to heat, light, and oxygen. They degrade due to oxidation and result in a rancid composition. Fish oils themselves have an unpleasant odor and flavor and are a liquid which makes them unacceptable for a number of *dry* foodstuffs such as powdered drink mixes, infant formula, health bars, breakfast cereals, baked goods, dressings and dairy products.

It is useful in terms of both storage and delivery, to encapsulate such oils in a free-flowing powder and enable the introduction of an otherwise liquid material into a powder, paste or cream formulation. Encapsulation can also enhance and prolong the active ingredients (enzymes) and functional characteristics of the encapsulated oils. Products containing oils that have been encapsulated are more easily processed and packaged and retain their functional characteristics for a longer shelf-life because the encapsulated oil is protected from degradation by other ingredients in the compositions as well as from atmospheric degradation. Encapsulation is also used to provide sustained release of the oils encapsulated.

There has been a continuing search for methods and compositions that effectively and

efficiently encapsulate such oils. Now generally well-established in the art is the introduction of the oil into an aqueous slurry that is then spray-dried in an environment of temperatures generally in the range of 185 to 200 degrees centigrade or higher, in order to achieve a powdered condition. Other traditional methods of dehydration in the manufacture the starch encapsulates include rotary drying, horizontal vacuum rotary drying, drum drying, fluidized bed drying, microwave drying, dielectrical drying, spouted bed drying, impingement drying, flash drying, or superheated steam drying.

Exposure to such temperatures damages the oil's enzymes (active ingredients). Enzymes are the catalysts of biochemical reactions and are responsible for bringing about almost all of the chemical reactions in living organisms.

The need exists for a simple, more efficient and effective oil storage and delivery system that can be used to store and deliver oils in dry compositions.

## **SUMMARY OF THE INVENTION**

The present invention relates to a free-flowing encapsulated oil particle comprising microreticulated or microfibrillated microcrystalline cellulose such as Solka-Floc, an organic oil, a modified starch such as malto-dextrin (with a low dextrose equivalent), and a synthetic, amorphous precipitated silica (silicon dioxide).

The present invention also relates to a process for manufacturing the above described encapsulated oil particle comprising the steps of dispersing microcrystalline cellulose into the oil within a commercial blender, blending, subsequently dispersing a modified starch, blending, and subsequently introducing, dispersing and blending a synthetic, amorphous precipitated silica (silicon dioxide). The resulting encapsulated oil particles are then milled to the desired particle size.

The present invention solves the long-standing need for a simple, cost effective,

storage-stable oil delivery system that preserves and protects the active ingredients of the oil. Further, encapsulated oil-containing compositions have reduced product odor during storage of the composition. The present invention also yields substantial monetary savings in the dehydration process.

All percentages, ratios, and proportions herein are on a weight basis unless otherwise indicated. All documents cited are hereby incorporated by reference in their entirety.

## **DETAILED DESCRIPTION OF THE INVENTION**

This invention relates to an encapsulated oil particle comprising a) a microreticulated or microfibrillated microcrystalline or powdered cellulose such as Solka-Floc, b) an oil, c) a modified starch such as malto-dextrin (with a low dextrose equivalent, preferably 10 or lower); and a synthetic, amorphous precipitated silica (silicon dioxide) such as Sipernat. The dispersion of each successive component is blended into the whole for specified times. Such particles can be designed to enhance or prolong the functional characteristics of encapsulated oils. For example, substances naturally of a liquid character can be formulated into a powder, paste or cream formulation, more easily adapted for packaging or for practical utility, such as for sustained release of said compositions.

The oil encapsulated by the present invention can be any oil that is a liquid between about 10.degree. C. and 90.degree. C. To prepare the encapsulated oil particle, the microcrystalline cellulose is first dispersed uniformly in the selected oil by blending 30 to 60 seconds. Immediately thereafter modified starch is dispersed uniformly in the mixture by blending 30 to 60 seconds. Immediately thereafter synthetic, amorphous precipitated silica (silicon dioxide) is dispersed uniformly in the mixture by blending 30 to 60 seconds. Particle inflation or "ballooning" effects are reduced because particle temperature remains low to yield a denser particle. Too much particle inflation leads to fracture of the encapsulate resulting in

poor physical stability.

While not wishing to be bound by theory it is believed that the absence of heat acts to decrease the vapor pressure of the oil during processing so that the oil has less of a tendency to migrate toward the surface of the capsule where it could be subsequently exposed to atmospheric influences. Additionally, the addition of "coating" effect of the silicon dioxide additive increases the amount of energy necessary to draw the oil out of the particle. Typically, without direct addition of silicon dioxide, a resulting capsule has surface oil amounting to approximately 2.0%. Surface oil is measured by extraction of the encapsulated particle with hexane at 25.degree. C. and atmospheric pressure, followed by gas chromatography. The hexane extracts only the oil on the surface of the particle, not the oil encapsulated within the particle. With the direct addition of a silicon dioxide to the oil, surface oil of the resultant encapsulated oil particle is reduced to below 0.1%. This improves the physical stability of the particles. Improved physical stability relates to less oil loss over time from the encapsulated oil particle.

The final process step involves "milling" to achieve the desired particulate size. The preferred embodiment calls for such milling to take place in a cold chamber mill in order to reduce friction-induced heat.

The resulting encapsulated oil particles can also be described as agglomerates of the component ingredients in a dry, particulate form. By the term "agglomerate", as used herein, is meant a stable, substantially physical mixture of at least two components in its dry state whose components are loosely bound to each other when dried, but disperse into its component parts when reconstituted in water under typical food processing conditions. This physical state is to be distinguished over "aggregates", which are firmly bound components in particle form that

remain bonded to each other even if reconstituted in water under typical food processing conditions.

Encapsulation of oils as described above allows for loading of larger amounts of oil than if they were encapsulated in a native starch granule. Absorption of oils using cyclodextrin is limited by the particle size of the guest molecule (oil) and the cavity of the host (cyclodextrin). Traditional cyclodextrin molecules trap the oil completely inside their cavity thereby limiting the size and amount of the oil encapsulated. It is difficult to load more than about 20% oil into a cyclodextrin particle. However, encapsulation with a starch that has been modified to have emulsion properties does not impose this limitation. Since the encapsulation in the present invention is achieved by entrapping oil droplets of less than 15 microns, preferably less than 5 microns and most preferably less than 2.5 microns in size, within a modified starch matrix, more oil can be loaded based on the type, method and level of modification of the starch. Encapsulation with the modified starches described by this invention allows loads much greater than 20% oil.

Other suitable matrix materials and process details are disclosed in, e.g., U.S. Pat. No. 3,971,852, Brenner et al., issued Jul. 27, 1976, which is incorporated herein by reference.

It will be understood by those skilled in the art that they may make substitutions of chemical or physical equivalents to the above cited components. While such substitutions represent a less preferred embodiment of the current invention, all are deemed to be protected hereunder.